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Post-Remediation Biomonitoring of Pesticides in Marine Waters Near the United Heckathorn Superfund Site, Richmond, California

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Prepared for the
U.S. Environmental Protection Agency
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POST-REMEDATION BIOMONITORING OF
PESTICIDES IN MARINE WATERS NEAR THE
UNITED HECKATHORN SUPERFUND SITE,
RICHMOND, CALIFORNIA

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Pacific Northwest National Laboratory
Richland, Washington

SUMMARY

This report, PNNL-11911 Rev. 1, was published in July 2000 and replaces PNNL-11911, which was published in September 1998. The revision corrects tissue concentration units that were reported as dry weight but were actually wet weight, and updates conclusions based on the correct reporting units.

Marine sediment remediation at the United Heckathorn Superfund Site was completed in April 1997. Water and mussel tissues were sampled in January 1998 from four stations near Lauritzen Canal in Richmond, California, for the first post-remediation monitoring of marine areas near the United Heckathorn Site. Dieldrin and DDT were analyzed in water samples, tissue samples from resident mussels, and tissue samples from transplanted mussels deployed for 4 months. Concentrations of dieldrin and total DDT in water and total DDT in tissue were compared to pre-remediation data available from the California State Mussel Watch program (tissues) and the Ecological Risk Assessment for the United Heckathorn Superfund Site (tissues and water).

Chlorinated pesticide concentrations in water samples were similar to pre-remediation levels and did not meet remediation goals. Mean dieldrin concentrations in water ranged from 0.65 ng/L to 18.1 ng/L and were higher than the remediation goal (0.14 ng/L) at all stations. Mean total DDT concentrations in water ranged from 0.65 ng/L to 103 ng/L and exceeded the remediation goal of 0.59 ng/L. The highest concentrations of both pesticides were found in Lauritzen Canal, and the lowest levels were from the Richmond Inner Harbor Channel water. Unusual amounts of detritus in the water column at the time of sampling, particularly in Lauritzen Canal, could have contributed to the elevated pesticide concentrations and poor analytical precision.

Tissue samples from biomonitoring organisms (mussels) provide an indication of the longer-term integrated exposure to contaminants in the water column, which overcomes the limitations of a single-point-in-time water sample. Biomonitoring results indicated that pesticides were still bioavailable in the water column, and have not been reduced from pre-remediation levels. Total DDT and dieldrin concentrations in mussel tissues were generally higher than measured levels from pre-remediation surveys. Mean chlorinated pesticide concentrations were highest at Lauritzen Canal/End (4,003 µg/kg total DDT and 232 µg/kg dieldrin, wet weight; mean of resident and transplant mussels), followed by Lauritzen Canal/Mouth (1335 µg/kg total DDT and

103 µg/kg dieldrin, wet weight) and Santa Fe Channel/End (435 µg/kg total DDT and 45.6 µg/kg dieldrin, wet weight).

The lowest levels were found at the Richmond Inner Harbor Channel station (120 µg/kg total DDT and 8.3 µg/kg dieldrin, wet weight; mean of resident and transplant mussels). The relative increase above pre-remediation levels was greatest at the Richmond Inner Harbor Channel station, where post-remediation DDT concentrations were about 3 times higher than pre-remediation DDT concentrations.

Active dredging in the Santa Fe Channel may have contributed to the bioavailability of pesticides to mussels and the relative difference between pre- and post-remediation tissue concentrations, especially outside of Lauritzen Canal. Differences in tissue concentrations between resident and transplanted mussels were observed, but these were attributable, in part, to differences in lipid content of tissues. Either resident or transplanted mussels would be appropriate for biomonitoring in the future; both will be measured for at least one more year of the Heckathorn post-remedial monitoring program. Annual biomonitoring will continue to assess the effectiveness of remedial actions at the United Heckathorn Site.

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1.0 INTRODUCTION

The United Heckathorn Site is located in Richmond Harbor, on the east side of San Francisco Bay in Contra Costa County, California (Figure 1.1). The Site is an active marine shipping terminal operated by the Levin Richmond Terminal Corporation. The Site was listed by the U.S. Environmental Protection Agency (EPA) on its National Priorities List of Federal Superfund sites because of chemical contamination of upland and marine sediments. A Remedial Investigation of adjacent marine areas revealed widespread sediment contamination with pesticides, particularly DDT and dieldrin (White et al., 1994). Significant pesticide contamination was limited to the soft geologically recent deposits known as younger bay mud. Pesticide concentrations were highest in the Lauritzen Canal, and decreased with increasing distance from the former United Heckathorn Site, clearly indicating that Heckathorn was the source of contamination. An ecological risk assessment at the Heckathorn Site (Lee et al., 1994) reviewed data collected in 1991 and 1992 for contaminant concentrations in marine water, organisms, and sediments. This assessment revealed that DDT and dieldrin contamination, originating from the United Heckathorn Site, was actively transported to offsite areas via surface waters.

Tissue samples from mussels collected near the Lauritzen Canal have been analyzed for DDT and dieldrin in two previous studies. The Heckathorn Ecological Risk Assessment (Lee et al., 1994) analyzed tissues from native (i.e., resident) mussels collected from stations of opportunity in 1991 and 1992 in the Lauritzen Canal (ferry rudder and rope NE of ferry), the Santa Fe Channel (boat house float), and Richmond Inner Harbor Channel (red nun buoy #16). For the California State Mussel Watch program, transplanted mussels (*Mytilus californianus*) were deployed at four Mussel Watch sites in or adjacent to the United Heckathorn Superfund Site (Santa Fe Channel/Mouth, Santa Fe Channel/End, Lauritzen Canal/End, and Lauritzen Canal/Mouth) (Rasmussen 1995). The State Mussel Watch program's Santa Fe Channel/Mouth site and the Heckathorn Ecological Risk Assessment's Richmond Inner Harbor Channel site were had similar locations. The pre-remediation Ecological Risk Assessment and State Mussel Watch data were the basis for comparison with the current post-remediation biomonitoring results.

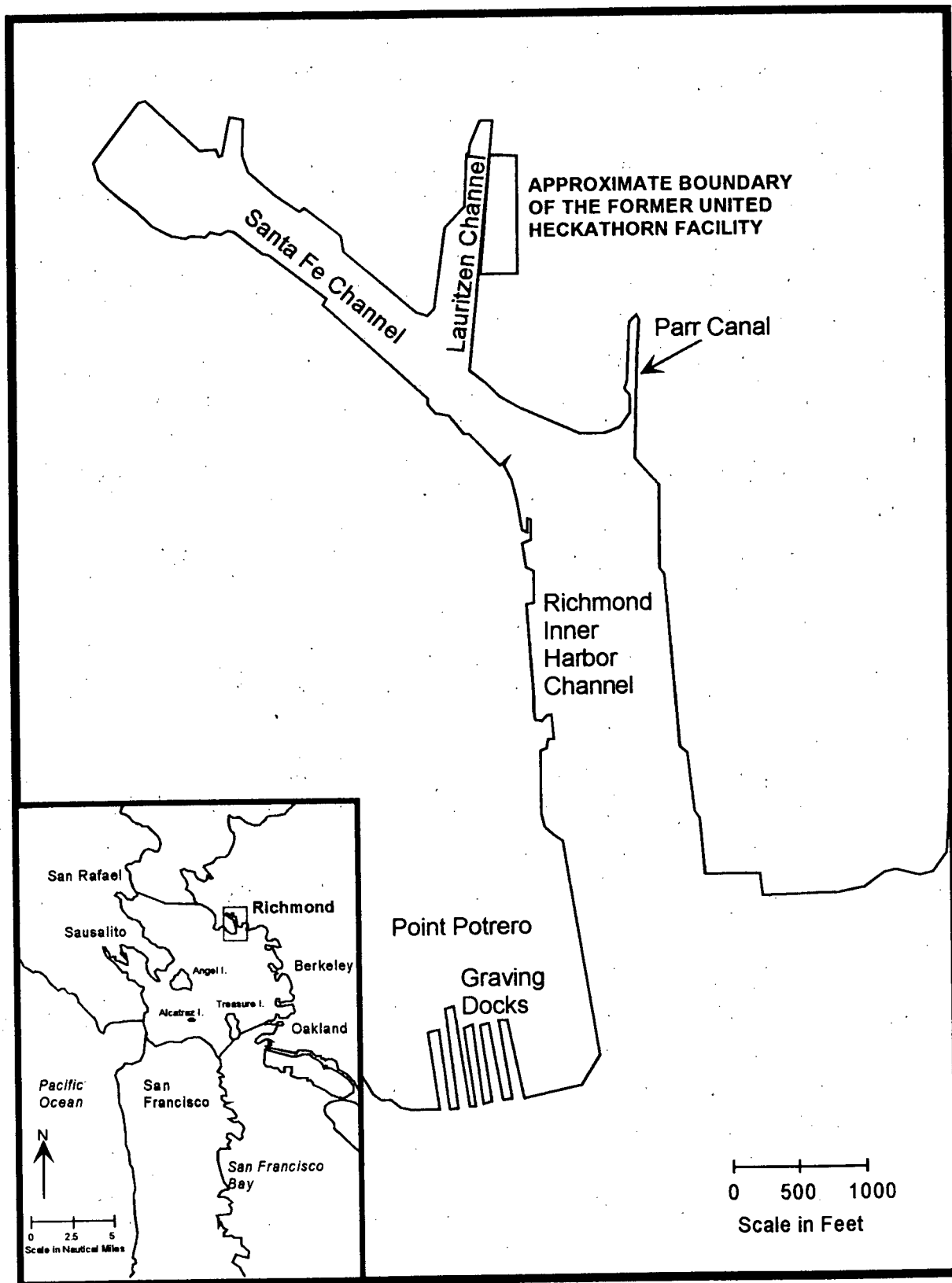


Figure 1.1. Location of the United Heckathorn Superfund Site, Richmond, California

The final remedial actions at the Heckathorn Site outlined in the Record of Decision (ROD 1996) have the following major components:

- dredging of all soft bay mud from the Lauritzen Canal and Parr Canal, with offsite disposal of dredged material,
- placement of clean material after dredging,
- construction of a cap around the former Heckathorn facility to prevent erosion,
- a deed restriction limiting use of the property at the former Heckathorn facility location to non-residential uses,
- marine monitoring to verify the effectiveness of the remedy.

Remediation levels that would be protective of the environment and human health were established to provide benchmarks for determining the effectiveness of the remedial actions. The Feasibility Study (Lincoff et al., 1994) and the ROD reviewed federal and state environmental laws that contained Applicable or Relevant and Appropriate Requirements (ARARs) for the remedial actions. EPA marine chronic and human health water quality criteria (WQC) were identified as ARARs for surface water. Because the human health standards based on consumption of contaminated fish are lower, these were selected as remedial goals. No chemical-specific ARARs were identified as remedial goals for marine sediments or tissues at the site.

This report provides the results of the first phase of post-remediation monitoring. The purpose of the marine monitoring is to demonstrate a reduction in flux of contaminants from the United Heckathorn Superfund Site following EPA response actions, including soil removals, dredging, and cap placement at the former Heckathorn facility. The measurement endpoints for this long-term monitoring are mussels and surface waters. Remediation levels set forth in the ROD are provided in Table 1.1.

Table 1.1. Remediation Levels for Surface Water Specified in the Record of Decision for the United Heckathorn Superfund Site

Chemical	DDT (total) ^(a)	Dieldrin
Remediation Goal	0.59 ng/L	0.14 ng/L
(a) The sum of the 4,4'- and 2,4'-isomers of DDT, DDD (TDE), and DDE		

2.0 METHODS

Methods for collection, processing, and analysis of tissue and water samples were outlined in the Field Sampling and Analysis Plan (Battelle 1997); a brief review is provided here. All procedures for sampling, sample custody and field/lab documentation, plus other aspects of documentation, quality assurance, and sample analysis were consistent with the Quality Assurance Project Plan (QAPjP) for Remedial Investigation and Feasibility Study of Marine Sediments at the United Heckathorn Superfund Site (Battelle 1992).

The four post-remedial monitoring stations were selected to overlap stations sampled in the pre-remedial State Mussel Watch program (Figure 2.1). Three of the stations also overlap with locations sampled during the Ecological Risk Assessment (Lee et al., 1994). The Ecological Risk Assessment Lauritzen Canal station corresponds to Mussel Watch Station 303.3, Lauritzen Canal/End; the Ecological Risk Assessment Santa Fe Channel station corresponds to Mussel Watch Station 303.4. The Richmond Inner Harbor station for the Ecological Risk Assessment was at the navigational nun buoy (#16), which is about 1200 ft offshore from Richmond Inner Harbor Channel. There was no Ecological Risk Assessment sampling station that corresponded with Mussel Watch Station 303.2, Lauritzen Canal/Mouth. Mussel tissue samples were collected and analyzed in both of the pre-remedial studies, but no water samples were analyzed for the State Mussel Watch program. A more detailed description of sampling stations is provided in the Field Sampling Summary memo (Appendix A; Lincoff 1998).

2.1 Collection and Deployment of Transplanted Mussel Stock

California mussels (*M. californianus*) were collected on September 3, 1997, from the north side of Bodega Head, California. This is the same area used by the California State Mussel Watch program for collection of transplant mussel stock (Gary Ichikawa, California Department of Fish and Game, personal communication). Scientists from the EPA Region 9 laboratory and Battelle Marine Sciences Laboratory (MSL) gathered mussels by hand at low tide from rocky intertidal habitat at approximately +1 ft to +3 ft mean lower low water (MLLW). Mussels were collected in nylon mesh bags and were held in coolers for transport.

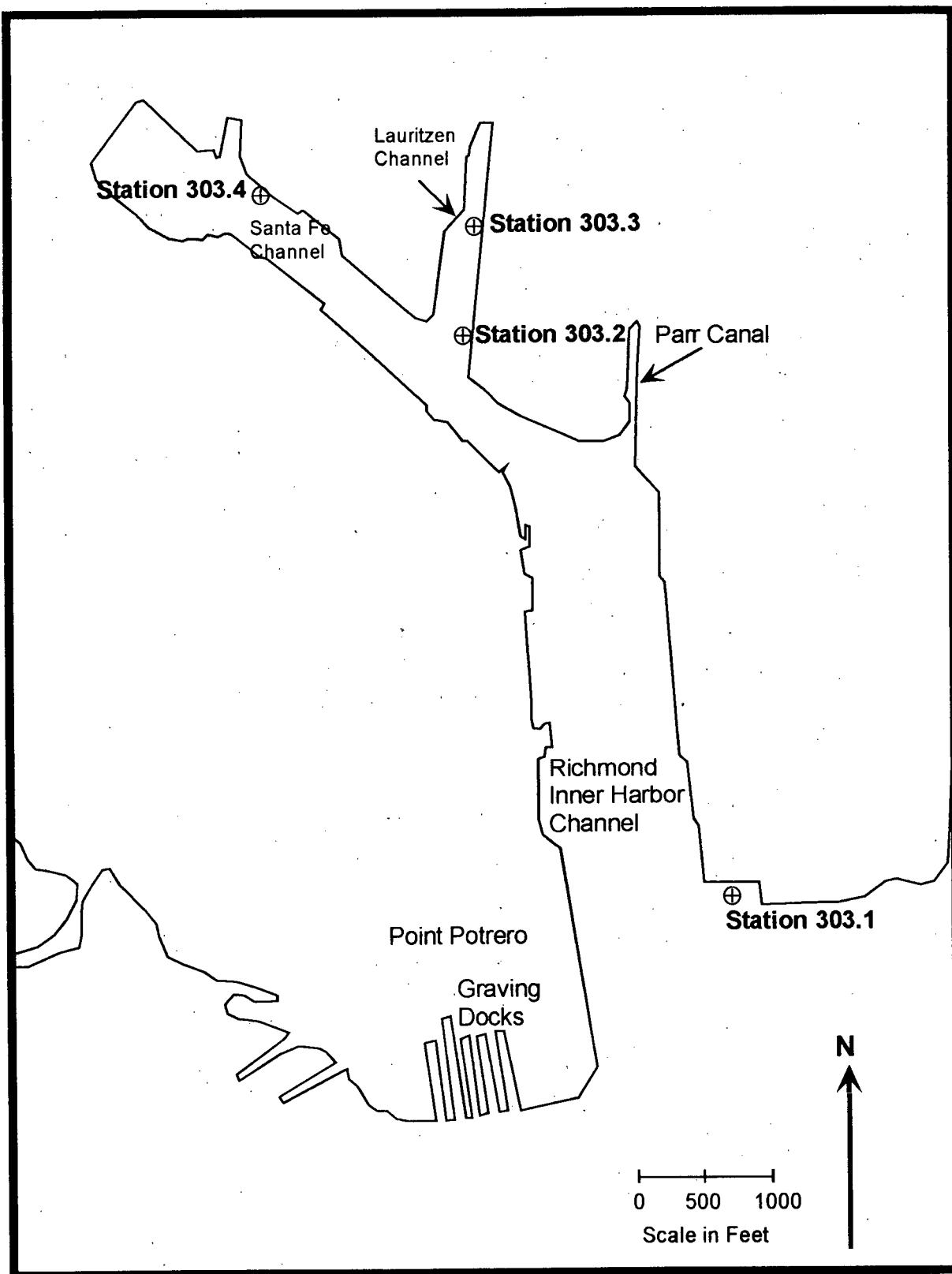


Figure 2.1. Sampling Stations for Long-Term Post-Remediation Monitoring of the United Heckathorn Site

At the EPA Region 9 laboratory, mussels were cleaned gently to remove epiphytes, and sorted to select individuals at approximately 40 mm to 60 mm shell length. Selected mussels were placed in tubular plastic mesh bags, divided into three groups of approximately 20 mussels each, and closed with a plastic cable tie. Mesh bags with transplanted mussels were tied to nylon rope and suspended subtidally at four sampling stations. Initial collection and deployment in the field was completed on the same day, September 3. Nylon ropes were placed inconspicuously to avoid vandalism.

2.2 Tissue and Water Sample Collection and Analysis

A background mussel tissue sample was prepared from the transplant mussel stock on the day of initial deployment (September 3, 1997). Approximately 45 whole mussels were placed in two layers of ashed aluminum foil, labeled, and packed in a sealed Ziploc bag. The sample was held on ice for transport to the analytical laboratory, then frozen and stored at -20°C until processed with other tissue samples in January 1998.

After transplanted mussels had been deployed for 4 months, seawater, transplanted California mussels (*M. californianus*), and resident blue mussels (*M. edulis*) were collected for analysis. On January 6, 1998, samples were collected at Richmond Inner Harbor Channel (Station 303.1), Lauritzen Canal/End (Station 303.3), and Santa Fe Channel/End (Station 303.4) (Figure 2.1). Resident blue mussels could have been one of several subspecies or hybrids in the *M. edulis* complex that cannot be easily distinguished by the shells alone (Harbo 1997). Samples were not collected on January 6 at Lauritzen Canal/Mouth (Station 303.2) because access to the transplanted mussels at this station was blocked by pile driving equipment at the Levin Pier. Safe access for sampling at Station 303.2 was available the next day, January 7, when samples were collected. Location coordinates presented in Table 2.1 were recorded for each station using a Global Positioning System with differential correction (dGPS). Samples were collected at low tide on a calm day with light rain. Ambient water temperature was 12°C. Water and tissue samples were also collected for analysis by the EPA Region 9 laboratory for an inter-laboratory comparison. A field sampling summary prepared by EPA Region 9 staff is provided here in Appendix A (Lincoff 1998).

Table 2.1. Sampling Stations for Post-Remediation Monitoring in 1997-1998 of the United Heckathorn Site

Station Number	Station Name	Location	Remarks
303.1	Richmond Inner Harbor Channel	37°54' 32.8" N 122°21' 34.5" W	On western most wooden dolphin southeast of public fishing pier
303.2	Lauritzen Canal/Mouth (South)	37°55' 12.6" N 122°22' 01.2" W	On east side directly across from concrete embankment/corner on western side
303.3	Lauritzen Canal/End (North)	37°55'22.5" N 122°21' 59.9" W	On east side, at small wooden pier
303.4	Santa Fe Channel/End	37°55' 21.53" N 122°21' 18.37" W	At west end of boat shed on north side, east of small boat fuel dock

Surface water samples were collected approximately 0.3 m below the water surface. To collect a sample, a bottle was submerged, the cap was removed under water to fill, and the cap replaced before the bottle was lifted. At each station, three 2-L water samples were collected for analysis by Battelle MSL. Additional water samples were collected for quality control (i.e., matrix spike, matrix spike duplicate, and blind duplicate water samples). Water samples were chilled to and held at 4°C until extracted. Water salinity was not recorded in the field but was measured in samples at the analytical laboratory. Salinity of all water samples checked pre-extraction was 28‰. Samples from Richmond Inner Harbor Channel and Lauritzen Canal/End were extracted before salinity was measured, and salinity in post-extraction water ranged from 32.5‰ to 34‰. Therefore, extraction seems to have altered the water salinity or refractory index to produce anomalous salinity values. Salinity of all water samples was assumed to have been 28‰.

Resident mussels were collected from approximately +1 ft MLLW at Richmond Inner Harbor Channel, Lauritzen Canal/Mouth, and Lauritzen Canal/End. Transplanted mussels had been deployed at approximately -3 ft MLLW at Richmond Inner Harbor Channel and Lauritzen Canal/Mouth, and at -6 ft MLLW at Lauritzen Canal/End. At Santa Fe Channel/End, resident and transplanted mussels were attached to a floating dock, under which resident mussels were within 1 ft of the water surface and transplanted mussels were approximately 8 ft below the water surface. Mussels were cleaned gently in the field and packaged whole in ashed foil and

plastic bags, as described above. Mussel samples were held frozen at -20°C until soft tissue samples were processed for analysis. To prepare tissue samples, mussels were partially thawed, the valve or shell length was measured, byssus threads were cut from the tissue, and soft tissues were transferred to a sample jar. Sand and mud on the soft tissue was rinsed off with deionized water. Each tissue sample was comprised of between 35 and 45 individual mussels. The total wet weight of each tissue sample was recorded. Tissue samples were re-frozen until extracted.

Chemical analyses followed methods described in the QAPjP (Battelle 1992). Water and tissues samples were analyzed for chlorinated pesticides. Tissue samples were also analyzed for total lipids. Total DDT was calculated as the sum of detected concentrations for six DDT compounds: 2,4-DDE, 4,4-DDE, 2,4-DDD, 4,4-DDD, 2,4-DDT, and 4,4-DDT. The detection limit was not used in calculation of total DDT. Total DDT, or sum of DDTs, was calculated in the same manner in the California State Mussel Watch program (Rasmussen 1995) and the Ecological Risk Assessment for the United Heckathorn Superfund Site (Lee et al., 1994).

3.0 RESULTS AND DISCUSSION

This section presents the results of physical measurements to assess the size and health of transplanted and resident mussels, and the results of pesticide analysis of water and mussel tissue. All extractions and analyses were conducted within target holding times. Complete data tables with results for water and tissue analyses, including quality control data, are provided in Appendix B. The current 1998 post-remedial water monitoring data are compared with pre-remedial data from the Ecological Risk Assessment and with the remedial goals for the site. The current post-remedial tissue monitoring data are compared with pre-remedial tissue concentrations from the State Mussel Watch program and the Ecological Risk Assessment.

3.1 Mussel Size and Health

Mussels collected for tissue samples were of similar size, although some individuals exceeded the preferred size range of 40 to 65 mm (combined ranges from Rasmussen 1995 and Lee et al., 1994). Raw data for shell length measurements and mean wet weight per mussel is provided in Table 3.1. Shell length of transplanted California mussels in the background sample ranged from 45 mm to 62 mm (mean = 52 mm). Four months later, California mussels transplanted to the study site were between 44 mm and 66 mm long (mean = 54 mm). Resident mussels collected in January 1998 ranged from 40 mm to 76 mm shell length (mean = 56 mm).

The overall mean weight of mussels was calculated as the total wet weight of the tissue sample divided by the number of individuals per sample. Mean weights of mussels were 5.28 g for the background sample, and 5.81 g and 5.84 g for transplanted and resident mussels in January 1998, respectively. These data showed that transplanted California mussels grew in both length and weight during the four-month deployment period.

The lipid content was similar for the background tissue sample (9.60% dry weight) and transplanted mussel samples collected in January 1998 (range of 8.71% to 10.1%, mean of 9.60% dry weight). This indicated that the deployed transplanted mussels were in good health and that bioaccumulation of contaminants was not likely to have been compromised by poor health, poor water quality, or limited food availability for the deployed organisms. Lipid content of resident mussels was similar to but slightly more variable than that of transplanted mussels, ranging from 6.26% to 10.2% dry weight (mean of 8.99%). It should be noted that tissue lipid content is not a definitive indicator of organism health because lipid content in bivalves can vary significantly depending on the availability of food and the bivalve's reproductive cycle.

3.2 Water

Triplicate water samples were collected at each site on the same day. These samples provided a "snapshot" of water column concentrations of DDT compounds and dieldrin, but they provided no information about the temporal variability or vertical stratification of these contaminants in the water column or the variability in water column concentrations to which biomonitoring organisms had been exposed. The absence of evaluation of temporal variability should be considered when these data are compared with results from earlier studies. Pre-remediation water samples collected for the Ecological Risk Assessment (Lee et al., 1994) provided more data on temporal variability because samples were taken over three successive days at two different sampling periods, approximately four months apart.

Water samples were extracted with solvent, and solvent extracts were concentrated to 0.2-mL volume for an overall enhancement factor of approximately 10,000 in an attempt to achieve detection levels below the remediation goals. Recoveries of surrogate compounds were low in 6 of 15 water samples and 4 of 7 quality control samples because of the additional drying steps required to remove residual water, potential loss of portions of samples, and extra evaporation steps necessary to achieve a low final sample volume. All data were corrected using the

Table 3.1. Length and Weight Data from Mussels Collected for Tissue Samples in January 1998 for Post-Remediation Monitoring of the United Heckathorn Site

Mussel #	Shell Length (cm)								Background
	303.1		303.2		303.3		303.4		
	Transplant	Resident	Transplant	Resident	Transplant	Resident	Transplant	Resident	
1	5.20	5.18	5.99	6.86	5.77	6.75	4.71	6.49	5.50
2	6.02	6.23	5.05	6.90	5.13	6.00	5.26	6.12	5.48
3	5.20	5.04	5.52	7.55	5.49	5.96	4.99	6.41	4.85
4	5.15	5.85	4.81	6.85	5.39	4.53	4.40	5.69	6.10
5	5.54	5.59	4.79	5.96	6.39	4.75	5.25	5.26	4.50
6	5.52	6.41	5.43	6.50	5.16	4.65	5.55	5.90	5.53
7	5.35	5.49	5.07	6.74	5.08	4.66	4.86	5.89	5.67
8	5.80	5.78	6.29	5.91	5.79	5.19	4.85	5.76	5.26
9	5.47	4.90	4.80	6.11	5.66	5.80	5.94	5.88	5.81
10	5.38	5.65	5.26	6.49	5.50	4.65	5.40	6.03	4.73
11	5.56	5.95	5.27	4.98	4.96	6.92	5.17	5.16	5.61
12	5.01	5.85	6.40	5.63	5.25	4.71	6.05	5.21	4.84
13	5.43	5.61	4.58	7.07	4.90	5.07	5.53	5.24	5.24
14	4.98	5.22	5.17	4.59	5.98	5.52	5.05	5.55	5.15
15	4.72	5.51	5.03	5.68	6.58	4.81	5.12	6.32	5.10
16	5.68	6.90	4.86	7.29	5.76	4.25	5.04	5.87	4.91
17	5.06	5.51	5.01	6.21	4.70	4.94	5.47	6.17	5.65
18	6.07	6.17	4.89	5.35	5.94	6.50	6.08	5.68	4.92
19	4.89	5.15	6.24	6.21	5.53	5.50	5.27	5.75	5.14
20	4.92	4.75	5.00	6.32	5.90	5.42	5.13	5.49	4.68
21	5.23	5.79	5.05	5.97	5.15	6.29	5.76	4.55	5.02
22	6.57	6.75	5.00	5.86	4.74	6.54	6.00	5.22	5.08
23	5.33	6.95	5.01	5.00	5.77	6.59	4.84	6.40	5.62
24	4.94	6.01	6.00	5.49	5.65	6.61	5.87	4.98	4.78
25	5.69	5.15	4.92	6.00	5.67	6.38	5.09	5.95	5.23
26	5.05	5.07	5.44	6.14	6.12	5.63	5.91	6.25	5.47
27	5.72	6.56	6.23	6.14	5.17	4.94	4.84	6.32	5.09
28	5.50	6.01	4.82	5.88	5.80	4.94	5.48	4.81	5.51

Table 3.1. (contd)

Mussel #	Shell Length (cm)								Background
	303.1		303.2		303.3		303.4		
	Transplant	Resident	Transplant	Resident	Transplant	Resident	Transplant	Resident	
29	5.38	4.18	6.36	5.66	6.28	4.28	5.11	5.26	5.35
30	5.20	6.49	4.72	5.72	5.78	4.65	5.20	6.20	5.12
31	5.36	4.13	4.65	6.32	5.26	4.26	5.59	5.81	4.97
32	5.36	5.05	5.76	6.12	5.18	5.14	5.55	5.99	5.66
33	5.31	5.05	5.56	5.21	5.50	4.86	5.41	5.10	6.15
34	5.65	4.58	6.05	5.57	5.39	5.53	4.88	6.27	5.04
35	5.07	6.33	6.05	6.18	6.31	6.44	5.54	5.31	5.15
36		6.16	5.11	5.75	5.72	5.62	5.93	5.38	4.68
37		5.87	5.57	5.19	6.14	4.91	6.33	5.37	4.68
38		5.11	5.61	5.45	5.01	4.00	6.03	5.22	4.85
39		5.30	4.87	5.53	5.42	5.12	5.26	5.05	5.47
40		5.94	5.61	6.25	5.76	4.65	4.90	5.98	5.04
41		5.88	4.93	5.22	6.29	4.37	5.18	4.90	4.66
42		6.91	5.86	6.05	5.41	5.77	4.80	6.31	4.95
43		5.78	5.07	5.71	5.41	4.53		5.36	5.21
44		5.48	5.35	5.57	5.62			4.99	
45		5.34	4.59	4.74	6.04			4.77	
minimum length (cm)	4.72	4.13	4.58	4.59	4.70	4.00	4.40	4.55	4.50
maximum length (cm)	6.57	6.95	6.40	7.55	6.58	6.92	6.33	6.41	6.15
mean length (cm)	5.37	5.66	5.32	5.91	5.59	5.27	5.37	5.60	5.18
mean weight per mussel (g wet)	5.67	5.92	5.37	7.69	7.01	4.35	5.20	5.40	5.28
mean length (cm)		background	5.18	resident	5.61	transplants	5.41		
mean weight (g wet)		background	5.28	resident	5.84	transplants	5.81		

PCB 198 surrogate percent recovery. The achieved detection limits in water samples ranged from 0.01 ng/L to 0.11 ng/L for the six DDT compounds. Recovery of one of two DDT matrix spikes was 129%, slightly exceeding the quality control limits of 40%-120%. In the method blank, 0.11 ng/L DDE was detected; samples with <5X the blank concentration are flagged with a "B". Replicate precision was generally poor for the cleanest station where levels were low and for the most contaminated station where organic detritus could have been a contributing factor. Given these quality control concerns, the results of water analyses should be considered estimates.

Concentrations of DDT and dieldrin measured in 1998 post-remedial water samples are shown in Table 3.2. Water column concentrations of total DDT and dieldrin were similar in the 1991-1992 pre-remediation and 1998 post-remediation studies (Table 3.3), except at Station 303.3 Lauritzen Canal/End. Total DDT was higher at Lauritzen Canal/End in post-remediation water samples (80 ng/L to 138 ng/L, mean 103 ng/L) than in pre-remediation water samples (22 ng/L to 68 ng/L total DDT, mean 50 ng/L).

Table 3.2. Concentrations of DDT and Dieldrin in Water Samples Collected in January 1998 for Post-Remedial Monitoring of the United Heckathorn Site

Water Sample ID	Repli- cate	Location	Analyte and Concentration (ng/L)							Total DDT
			Dieldrin	2,4 DDE	4,4 DDE	2,4 DDD	4,4 DDD	2,4 DDT	4,4 DDT	
303.1	1	Richmond	0.47	0.02 U	0.15 B	0.28	0.05 U	0.05 U	0.04 U	0.43
303.1	2	Inner Harbor	1.16	0.02 U	0.17 B	0.44	0.05 U	0.05 U	0.04 U	0.61
303.1	3	Channel	0.32	0.03 U	0.17 B	0.10 U	0.52	0.22	0.04 U	0.91
303.2	1	Lauritzen	8.84	0.02 U	2.25	6.60	19.7	3.90	12.6	45
303.2	2	Canal	10.3	0.02 U	2.24	6.31	18.6	3.69	15.6	46
303.2	3	Mouth	5.40	0.02 U	2.34	6.06	14.8	3.72	9.33	36
303.3	1	Lauritzen	14.7	0.37	8.57	16.7	31.3 D	7.79	27.0 D	91
303.3	2	Canal	22.5	0.39	15.7	17.6	27.8 D	10.6	65.9 D	138
303.3	3	End	17.1 D	1.07	43.5 D	6.98	2.28	7.80	19.1	80
303.4	1	Santa Fe	2.65	0.02 U	0.03 U	2.01	9.62	0.10	0.04 U	12
303.4	2	Channel	2.16	0.02 U	0.03 U	2.04	8.07	0.15	0.04 U	10
303.4	3	End	2.61	0.02 U	0.03 U	2.26	8.84	0.05 U	0.04 U	11

U Not detected at or above DL shown
 B Analyte detected in sample is <5x blank concentration
 D 5X Dilution

Table 3.3. Comparison of 1998 Mean Post-Remediation Total DDT and Dieldrin in Water with Pre-Remediation and Remediation Goal Concentrations (all concentrations ng/L)

Water Sample ID	Location	1998 Post-Remediation		Remediation Goals		Pre-Remediation ^(a)	
		Total DDT	Dieldrin	Total DDT	Dieldrin	Total DDT	Dieldrin
303.1	Richmond Inner Harbor Channel	0.65	0.65	0.59	0.14	1	<1
303.2	Lauritzen Canal Mouth	42.6	8.18	0.59	0.14	no sample	no sample
303.3	Lauritzen Canal End	103	18.1	0.59	0.14	50	18
303.4	Santa Fe Channel End	11.0	2.47	0.59	0.14	8.6	1.8

(a) Pre-remediation water concentration is average of samples collected in October 1991 and February 1992 for the Ecological Risk Assessment (Lee et al., 1994).

As stated above, post-remediation water samples represent contaminant concentrations at a single point in time. It is impossible to determine from these data if the high levels of DDT in this post-remediation study represent a persistent condition or were a temporary event, perhaps caused by suspension of sediment from the nearby pile driving activity. In addition, an organic surface scum and detritus in the water column at the time of sampling in 1998, particularly at Lauritzen Canal/End (Station 303.3), could have contributed to elevated pesticide levels and greater field replicate variability.

Water concentrations of dieldrin and total DDT were well above remediation goals in all water samples, with one exception (Table 3.3); total DDT in water from Richmond Inner Harbor Channel was detected at levels near the remediation goal of 0.59 ng/L. Triplicate water samples at this station ranged from 0.43 ng/L to 0.91 ng/L total DDT, with a mean of 0.65 ng/L. Dieldrin in water from Richmond Inner Harbor Channel, however, was approximately 5 times higher than the remediation goal (0.14 ng/L). The most elevated contaminant concentrations were found in Lauritzen Canal water, where total DDT levels were between 72 times (Lauritzen Canal/Mouth) and 175 times (Lauritzen Canal/End) greater than the remediation goal. Dieldrin levels at Lauritzen Canal stations were 58 times and 129 times higher than the remediation goal. At the Santa Fe Channel/End, both total DDT and dieldrin were approximately 18 times higher than remediation goals.

3.3 Tissues

Tissue samples from biomonitoring organisms provide a time-integrated indication of contaminant concentrations in the water column. These values, therefore, overcome the limitations associated with sampling water on a single day. For tissue sample analysis, all quality control requirements were met. Achieved detection limits ranged from 0.15 µg/kg to 0.52 µg/kg wet weight. The background tissue sample had 1.05 µg/kg total DDT and 0.66 µg/kg dieldrin (wet weight). Results of tissue analyses (in wet weight) from transplanted and resident mussels are provided in Table 3.4.

The 1998 post-remediation data are compared with pre-remediation data in Table 3.5. Post-remediation levels of total DDT were lowest at the Richmond Inner Harbor Channel station, at 127 µg/kg in resident mussels and 113 µg/kg in transplanted mussels. At the Santa Fe Channel/End station, total DDT was 256 µg/kg in resident mussels and 613 µg/kg transplanted mussels. At the Lauritzen Canal/Mouth, total DDT was 1222 µg/kg in resident and 1448 µg/kg in transplanted mussels. The highest levels were found in mussels from Lauritzen Canal/End, in which total DDT was 4504 µg/kg in resident and 3502 µg/kg in transplanted mussels. Trends for dieldrin in mussel tissues were similar, with the highest levels at Lauritzen Canal/Mouth and Lauritzen Canal/End (103 µg/kg and 232 µg/kg dieldrin; mean of transplanted and resident mussels, respectively) and the lowest levels at the Richmond Inner Harbor Channel station (mean of 8.3 µg/kg dieldrin).

Tissue burdens from the Lauritzen Canal stations in this first post-remediation survey remained elevated, at higher levels than pre-remediation tissue burdens (Table 3.5). Resident mussels within Lauritzen Canal were exposed to suspended sediment during remediation, but the high levels in the transplanted mussels indicated that DDT was bioavailable in the 9 months following remediation as well. In Lauritzen Canal/End mussels, the average post-remediation DDT burden was 1.4 times higher than the pre-remediation (Ecological Risk Assessment) DDT burden. Tissue burdens in stations outside Lauritzen Canal showed an even higher relative difference between post- and pre-remediation levels, with post-remediation Richmond Harbor mussels containing about 3 times as much total DDT as pre-remediation mussels from the same location (Table 3.5). Richmond Harbor Channel and Santa Fe Channel were deepened between September 1997 and August 1998; mussels at all of the monitoring stations would have been exposed to suspended material during this operation.

Table 3.4. Concentrations of DDT and Dieldrin in Tissue Samples Collected in January 1998 for Post-Remediation Monitoring of the United Heckathorn Site

Analyte	Sample ID and Concentration (µg/kg wet weight)								
	Background ^(a)	303.1 Richmond Inner Harbor Channel		303.2 Lauritzen Canal Mouth		303.3 Lauritzen Canal End		303.4 Santa Fe Channel End	
		Transplant	Resident	Transplant	Resident	Transplant	Resident	Transplant	Resident
2,4 DDD	0.25 U	17.2 D	20.4 D	272 D	203 DD	702 DD	820 DD	138 D	47.1 D
2,4 DDE	0.26 U	0.26 U	0.26 U	8.22 D	10.2 D	19.5 D	29.5 D	4.46 D	3.03 D
2,4 DDT	0.18 U	4.52	7.46	81.2 D	145 D	220 DD	549 DD	26.2 D	15.0 D
4,4 DDD	0.38	70.5 D	66.4 D	825 DD	589 DD	1980 DD	1860 DD	349 DD	136 D
4,4 DDE	0.67	11.7 D	19.4 D	119 D	107 DD	207 DD	434 DD	54.2 D	29.9 D
4,4 DDT	0.15 U	9.04	13.6	143 D	168 DD	373 DD	811 DD	41.3 D	24.7 D
DIELDRIN	0.66	11.2 D	5.43	165 D	40.3 D	279 DD	184 D	83.1 D	8.18
Total DDT (wet wt)^(b)	1.05	113	127	1448	1222	3502	4504	613	256
Percent Dry Wt	15.2	11.8	10.8	10.8	8.90	10.9	10.6	11.9	8.94
Total DDT (dry wt)	6.91	957	1178	13411	13733	32124	42486	5153	2861
Dieldrin (dry wt)	4.34	94.9	50.3	1528	453	2560	1736	698	91.5
Lipids (%dry wt)	9.60	9.61	9.57	9.99	10.2	8.71	9.94	10.1	6.26
DDT (ppb lipid)	72.0	9961	12313	134247	134633	368816	427423	51016	45695
Dieldrin (ppb lipid)	45.2	988	525	15293	4439	29387	17463	6914	1462

D 9X Dilution

DD 100X Dilution

U Not detected at or above DL shown

(a) Background tissue concentration is from coastal *M. californianus* prior to deployment (transplanting) in Richmond Harbor.

(b) Total DDT is sum of detected 2,4- and 4,4- DDD, DDE, and DDT.

Table 3.5. Comparison of 1998 Post-Remediation Total DDT and Dieldrin in Tissues with Pre-Remediation Concentrations ($\mu\text{g/kg}$ wet weight)

Station Number	Station Name	State Mussel Watch ^(a)	Ecological Risk Assessment ^(b)	January 1998 Post-Remediation	January 1998 Post-Remediation
		Transplant	Resident	Resident	Transplant
<u>Total DDT (µg/kg wet weight)</u>					
303.1	Richmond Inner Harbor Channel	47.0 ^(c)	40	127	113
303.2	Lauritzen Canal/Mouth	629 ^(d)	---	1,222	1,448
303.3	Lauritzen Canal/End	5074 ^(d) 1369 ^(c)	2900	4,504	3,502
303.4	Santa Fe Channel/End	369 ^(c)	350	256	613
<u>Dieldrin (µg/kg wet weight)</u>					
303.1	Richmond Inner Harbor Channel	7.7 ^(c)	4	5.43	11.2
303.2	Lauritzen Canal/Mouth	87.0 ^(d)	---	40.3	165
303.3	Lauritzen Canal/End	602 ^(d) 100 ^(c)	97	184	279
303.4	Santa Fe Channel/End	32.5 ^(c)	19	8.18	83.1

(a) Most recent data available from State Mussel Watch program, transplanted California mussels (Rasmussen 1995).

(b) Average concentration in resident mussel tissue from samples collected in October 1991 and February 1992 (Lee et al., 1994).

(c) State Mussel Watch program sample from March 1991 (Rasmussen 1995).

(d) State Mussel Watch program sample from January 1988 (Rasmussen 1995).

A direct comparison of tissue burdens from different sampling dates can be confounded by differences in lipid content of tissues, because nonpolar organic compounds such as DDT tend to accumulate in fatty tissue. Lipid-normalized values for total DDT and dieldrin, expressed as μg pesticide/kg lipid weight, are provided in Table 3.6. For mussels collected in Lauritzen Canal, lipid-normalized tissue values confirmed an increase in DDT and dieldrin in mussels in

Table 3.6. Comparison of Lipid-Normalized 1998 Post-Remediation Total DDT and Dieldrin in Tissues with Pre-Remediation Concentrations ($\mu\text{g/kg}$ lipid weight)

Station Number	Station Name	State Mussel Watch ^(a)	Ecological Risk Assessment ^(b)	January 1998 Post-Remediation	January 1998 Post-Remediation
		Transplant	Resident	Transplant	Resident
<u>Total DDT (µg/kg lipid weight)</u>					
303.1	Richmond Inner Harbor Channel	9,215 ^(c)	3,275	9,961	12,313
303.2	Lauritzen Canal/Mouth	78,481 ^(d)	---	134,247	134,633
303.3	Lauritzen Canal/End	583,819 ^(d) 380,361 ^(c)	250,411	368,816	427,423
303.4	Santa Fe Channel/End	47,283 ^(c)	21,919	51,016	45,695
<u>Dieldrin (µg/kg lipid weight)</u>					
303.1	Richmond Inner Harbor Channel	1,507 ^(c)	322	988	525
303.2	Lauritzen Canal/Mouth	10,861 ^(d)	---	15,293	4,439
303.3	Lauritzen Canal/End	69,272 ^(d) 27,778 ^(c)	8,590	29,387	17,463
303.4	Santa Fe Channel/End	4,167 ^(c)	1,126	6,914	1,462

(a) Most recent data available from State Mussel Watch program, transplanted California mussels (Rasmussen 1995).

(b) Average concentration in resident mussel tissue from samples collected in October 1991 and February 1992 (Lee et al., 1994).

(c) State Mussel Watch program sample from March 1991 (Rasmussen 1995).

(d) State Mussel Watch program sample from January 1988 (Rasmussen 1995).

1998, relative to pre-remediation concentrations. Lipid-normalized tissue values for Santa Fe and Richmond Inner Harbor Channels showed slight increases in DDT and dieldrin relative to pre-remediation concentrations. Differences in lipid content did not account for the difference in dieldrin concentration observed between transplant and resident mussels: lipid-normalized

dieldrin concentrations were 1.7 to 4.7 times higher in transplanted mussels than those in resident mussels (Table 3.6).

Either transplanted or resident mussels appear to be acceptable for biomonitoring at the study site, but the differences between them should be monitored for at least one more year. Potential differences in total body burdens may have arisen from a variety of factors, including the use of different species, lipid content of tissues, duration of exposure, and height in the water column. Transplanted mussels were *M. californianus* that had negligible initial DDT contamination and that were exposed for a known time period at the study site (i.e., 4 months). Resident mussels were adult *M. edulis* that occur naturally at the study site, selected at approximately 40 mm to 60 mm shell length, that were likely to have been present before remediation had been completed. At all stations except Santa Fe Channel/End, the relative percent difference (RPD; difference/mean X 100) in total DDT between transplanted and resident tissue burdens in wet weight was 12% to 25%. At the Santa Fe Channel/End station, the RPD between resident and transplant tissue burdens was 82% (wet weight basis). Lipid-normalization reduced the apparent variability in tissue DDT burdens: the RPDs for total DDT on a lipid-normalized basis were between 0% and 21% at Richmond Inner Harbor and both Lauritzen Canal stations, and 11% at Santa Fe Channel/End. Thus, a portion of the difference between resident and transplanted mussels was attributable to differences in lipid content of tissues. Neither resident nor transplanted mussels were consistently higher or lower in total DDT concentrations.

For dieldrin, RPDs were higher than those for total DDT and ranged from 41% to 164% for wet weight data and from 51% to 130% for lipid-normalized data. Again, the biggest difference was observed at the Santa Fe Channel/End station. Dieldrin levels in resident mussels were consistently much lower than those in transplanted mussels (Tables 3.4, 3.5, and 3.6). Analysis of a background tissue sample confirmed that transplanted mussels did not contain significant levels of dieldrin at deployment. Therefore, it appears that transplanted mussels were exposed to higher dieldrin concentrations or were more effective in accumulating dieldrin than were resident mussels. This effect was not demonstrated for total DDT. Observed differences between transplanted and resident mussels also may have been attributable, in part, to height in the water column. At the Santa Fe Channel/End station, where the most significant difference in DDT burden between transplanted and resident mussels was found, mussels were attached to a floating boathouse. Resident and transplanted mussels were consistently at -0.5 ft and -8 ft below the water surface, respectively. The transplanted mussels were deployed at this depth to

reduce the probability of vandalism. Transplanted mussels at all other sites were attached to firm substrate at a fixed position. It is not known if attachment to a floating structure at different depths in the water column contributed to differences in bioaccumulation. In the future, it might be better to deploy mussels nearer to resident mussels and at a fixed height in the water column. In addition, sampling resident and transplanted mussels in early 1999 is recommended to provide more data for evaluation of differences in dieldrin uptake by the two types of mussels.

3.4 Conclusions

Results from the first post-remediation monitoring indicated that chlorinated pesticides remained in the Lauritzen Canal and in the semi-enclosed waters nearby. Remediation goals for total DDT and dieldrin in water have not yet been achieved for the study site. Biomonitoring indicated that the total DDT concentration in the water was not reduced, and in fact appeared to have increased somewhat, from pre-remediation levels in the first 9 months following remediation. As noted in the Field Sampling Summary, dredging for a deepening project in the Santa Fe and Richmond Inner Harbor Channels was ongoing since the fall of 1997, throughout most if not all of the time that transplanted mussels were deployed. Dredging operations started in the Santa Fe Channel, near Station 303.4, and was active near Brooks Island and Point Potrero, near Station 303.1, when samples were collected. Dredging activity was likely to have resuspended sediment containing DDT and dieldrin and may have influenced the water column concentration and potential exposure of mussels to these contaminants of concern. Unusual amounts of detritus in the water could also have contributed to elevated surface water measurements that are inconsistent with the mussel tissue results. Further biomonitoring will be important to determine if these data are representative of long-term bioavailability of pesticides from the Lauritzen Canal sediments.

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APPENDIX A
FIELD SUMMARY REPORT

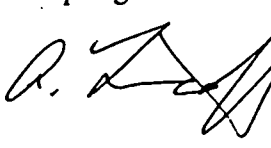


UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX LABORATORY
1337 S. 46TH STREET
BLDG 201
RICHMOND, CA 94804-4698

January 13, 1998

MEMORANDUM

SUBJECT: Summary of United Heckathorn Post-Remedial
Mussel and Surface Water Sampling

FROM: Andrew Lincoff, PMD-2 
Regional Laboratory

TO: Dick Vesperman, SFD-7-3
Remedial Project Manager

Attached is the Field Sampling Summary for the post-remedial mussel and surface water sampling at the United Heckathorn Superfund Site in Richmond, California. Transplanted California mussels were deployed at four locations in Richmond Harbor in September, 1997. On January 6 and 7, 1998, seawater samples, resident mussels and the transplanted mussels were collected. Samples were shipped to the Battelle Marine Sciences Laboratory in Sequim, Washington for analysis. Replicate samples were taken for analysis at the Regional Laboratory. Results are expected to be available in approximately two months and will be forwarded to you in separate reports.

If you have any questions, please call me at (510) 412-2330.

Attachment

✓ CC: LIAM ANTRIM

Field Sampling Summary for Mussels and Surface Water
at the United Heckathorn Site in
Richmond, California, conducted 1/6 - 1/7/98.

Andrew Lincoff
EPA Region 9 Laboratory
PMD-2
January 13, 1998

INTRODUCTION

This sampling event involved the collection of mussels and surface water samples from the Lauritzen Channel at the United Heckathorn Superfund Site and at other locations in Richmond Harbor in Richmond, California.

Sampling was performed by Andrew Lincoff and Amy Wagner of the EPA Region 9 Laboratory. Some of the mussels retrieved had been transplanted in September, 1997 with the assistance of Liam Antrim, of the Battelle Marine Sciences Laboratory, EPA's Superfund Program contractor.

Sampling was performed in accordance with Battelle's "United Heckathorn Post-Remediation Field Monitoring Plan" (FSP), dated February 5, 1997, with minor deviations discussed herein. The most significant change was that additional replicate samples were taken for analysis by the EPA Regional Laboratory in order to perform an inter-laboratory comparison to provide additional information regarding the accuracy of the results.

OBJECTIVE

EPA conducted this field sampling as part of the oversight of a final Remedial Action under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) at the United Heckathorn Site in Richmond, California. The sampling effort involved collecting physical environmental samples to analyze for the presence of hazardous substances.

The United Heckathorn Site was used to formulate pesticides from approximately 1947 to 1966. Soils at the Site and sediments in Richmond Harbor were contaminated with various chlorinated pesticides, primarily DDT, as a result of these pesticide formulation activities. The final remedy contained in EPA's October, 1994 Record of Decision addressed remaining hazardous substances, primarily in the marine environment. The major marine components of the selected remedy included:

- Dredging of all soft bay mud from the Lauritzen Channel and Parr Canal, with offsite disposal of dredged material.

- Marine monitoring to verify the effectiveness of the remedy.

The first component of the remedy selected in the ROD called for dredging all "young bay mud" from those channels in Richmond Harbor which contained average DDT concentrations greater than 590 ppb (dry wt.). The dredging was completed in April, 1997. The short-term monitoring, performed according to EPA's September 5, 1996 FSP, consisted of sediment chemistry monitoring to ensure that the average sediment concentration after dredging was below the cleanup level selected in the ROD. This monitoring was completed shortly prior to the placement of the sand cap in April, 1997.

Long-term monitoring is addressed by Battelle's February 5, 1997 FSP. The purpose of the long-term monitoring is to demonstrate the effectiveness of the remedy. Prior to the remediation, mussels in the Lauritzen Channel contained the highest levels of DDT and dieldrin in the State, and surface water exceeded EPA's Ambient Water Quality Criteria for DDT by a factor of 50. Lower but still elevated levels were found in mussels and surface water in the Santa Fe Channel. It was concluded in EPA's Remedial Investigation that these elevated levels were the result of continuous flux from contaminated sediments. Approximately 98% of the mass of DDT in sediments in Richmond Harbor was removed by the remedial dredging. The long-term monitoring will demonstrate whether this action has succeeded in reducing the levels of DDT in mussels and surface waters.

Battelle's FSP included monitoring using both transplanted California mussels and resident Bay mussels. The transplanted mussels were deployed in September and retrieved after approximately four months of exposure. The length of the deployment and seasonal timing were chosen to match the protocol used by the California State Mussel Watch Program, in order to permit comparison with the State's results over the past 15 years. Both transplanted and resident mussels will be analyzed to determine any difference. The results should be comparable. If the resident mussels have higher burdens, it may be due to past exposure. If the results are the same, only resident mussels will be collected in the future.

Laboratory results are expected from Battelle in approximately one month and will be provided in a report from Battelle in approximately two months. In addition, the EPA Regional Laboratory will perform an inter-laboratory comparison to provide additional information on the accuracy of the analyses. Replicates of the samples taken for analysis by Battelle will be analyzed by the Regional Laboratory with results expected in late March, 1998.

SAMPLE PLAN AMENDMENTS

The following deviations from the sample plan were made.

1. The FSP called for samples to be analyzed only by EPA's Superfund contractor, Battelle. In order to investigate the accuracy of the low-level seawater and tissue results, it was

decided to take additional sample volumes for analysis by the EPA Regional Lab in Richmond, California. These samples were taken at the same locations and at the same time as the Battelle samples.

2. The FSP called for ambient salinity measurements to be made during sampling. These were mistakenly not performed in the field, but will be performed by Battelle in the laboratory.

3. When the transplanted mussels were deployed in September 1997, a second set was hung beneath the Ford automotive plant for duplication in case of vandalism at Station 303.1. As none of the mussels were disturbed, the additional set (called 303.1X in the field log) was discarded.

FIELD NOTES AND OBSERVATIONS

1. Samples were taken on January 6 and 7, 1997 at low tide. The weather during the sampling was calm with clouds and occasional light rain. The ambient water temperature was 12 C at all sample locations.

2. Factors which may influence the results included ongoing dredging in Richmond Harbor and pier maintenance at the Levin Terminal in the Lauritzen Channel. The Richmond Harbor deepening project has been ongoing since the fall of 1997. The dredging started in the upper Santa Fe Channel, near site 303.4, and was near Brooks Island and Point Potrero when the samples were retrieved. The effect of the dredging during the mussel deployment is uncertain. The dredging probably resuspended sediment containing some DDT and dieldrin which could raise values. On the other hand, the dredging removed most of the remaining 2% of the mass of DDT from Richmond Harbor not removed by the Superfund Remedy. Thus the results could be lower than they would have been without the deepening project.

Another less likely potential influence was the replacement of piles at the Levin Pier during the retrieval of samples. Conceivably, the pile driving could have resuspended sediment beneath the pier and increased the pesticide load in mussels and seawater samples.

3. The sample station numbers, locations, date and times, and other information are shown in Figure 1 and listed in Table 1, below. Location coordinates were determined using GPS with differential correction. As discussed in the FSP, the station numbers are those used by the California Mussel Watch Program. Station 303.1 is at the entrance to the Richmond Inner Harbor Channel near the old Ford automotive plant. Mussels were deployed and collected from the western-most of the large dolphins near the plant. Station 303.2 is on the eastern side of the Lauritzen near its mouth. Mussels were deployed from pilings beneath the Levin Dock near the northern end of a large wooden fender structure. Station 303.3 is approximately 2/3 of the way up the Lauritzen Channel, on the eastern side. Mussels were hung from the southern end of a small wooden pier which extends out into the channel. This location is very close to where the highest levels of pesticide residues were removed from the Heckathorn Site. Station 303.4 is in

the upper Santa Fe Channel at the far western end of a large covered floating marina on the northern side.

Table 1
Mussel and Seawater Sample Locations

<u>Station</u>	<u>Date</u>	<u>Time</u>	<u>Location</u>	<u>Remarks</u>
303.1	1/6/98	1235	37 54' 32.8" N 122 21' 34.5" W	Richmond Channel
303.2	1/7/98	1245	37 55' 12.6" N 122 22' 01.2" W	Lauritzen South EPA QA/QC Mussels
303.3	1/6/98	1315	37 55' 22.5" N 122 21' 59.9" W	Lauritzen North Battelle Blind Dup. Seawater
303.4	1/6/98	1355	37 55' 21.53" N 122 21' 18.37" W	Santa Fe Battelle and EPA MS/MSD Seawater

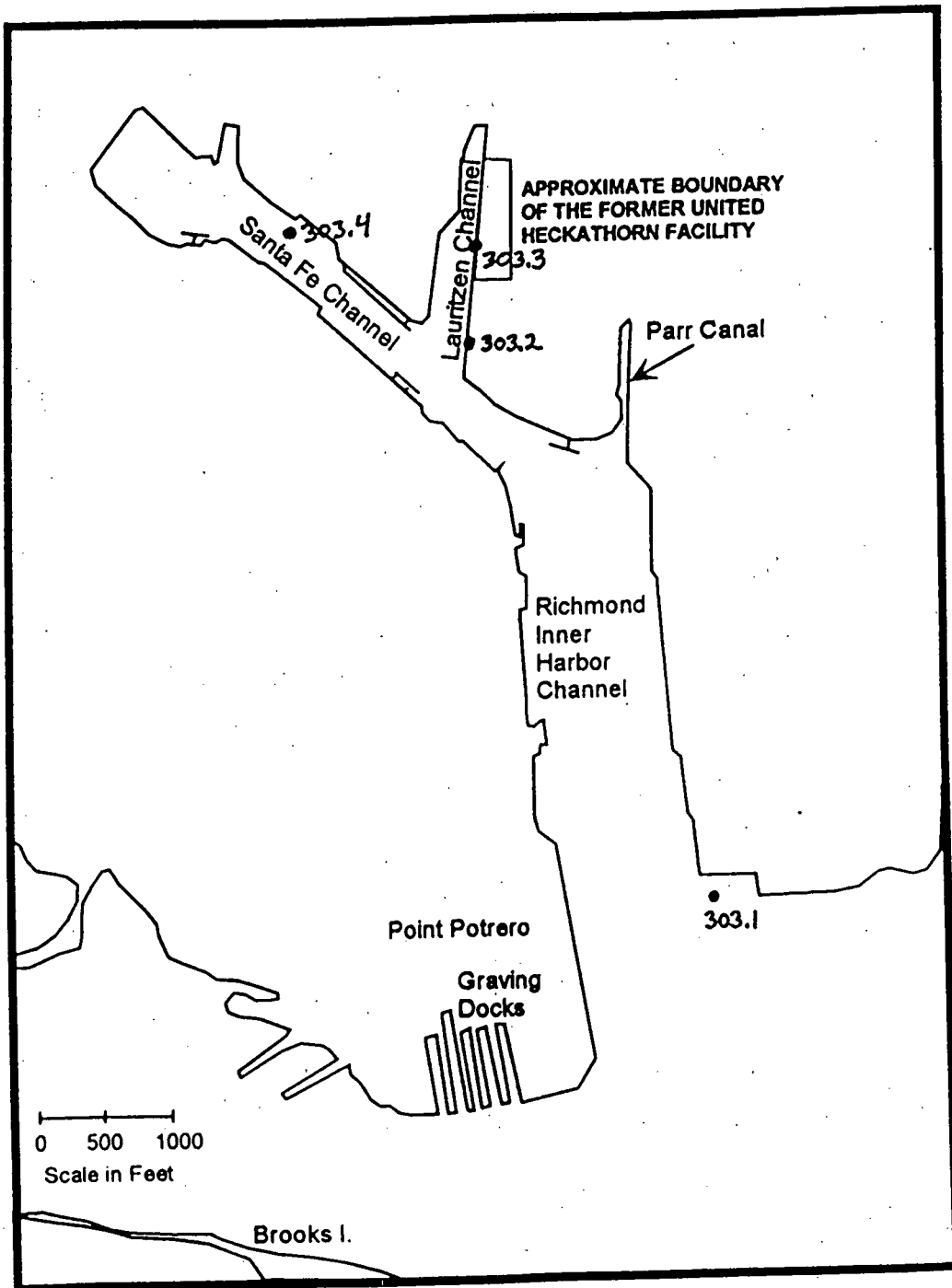
Seawater, transplanted California Mussels, and resident Bay mussels were collected at each station for analysis by Battelle and the EPA Regional Laboratory. At each station three 2 liter replicate seawater samples were collected for analysis by Battelle, and two 1 liter replicate seawater samples were collected for EPA. At station 303.4, two additional 2 liter seawater samples were collected for Battelle QA/QC, and two additional 1 liter samples were collected for EPA QA/QC. An additional single 2 liter blind duplicate of seawater sample 303.3 was collected and shipped to the Battelle Lab with the fictitious station number 303.5.

At each station, approximately 60 transplanted mussels and 60 resident mussels were collected. Approximately 45 of these were sent to Battelle and the remainder (approximately 15) will be analyzed by the EPA Regional Lab. The 45 mussels per sample sent to Battelle is large enough for any sample to be selected by Battelle for laboratory QA/QC. At station 303.2 additional resident mussels were collected so that a total of 27 were retained for the EPA Regional Lab. This sample was designated for EPA mussel QA/QC.

The resident mussels were all collected near the surface, which at the collection times and dates was approximately 1 foot above Mean Lower Low Water (+1 ft MLLW) for the samples collected from pilings at stations 303.1, 303.2, and 303.3. At station 303.4, the mussels were collected near the surface from a floating dock. The transplanted mussels were deployed at the following approximate depths: 303.1, -3 ft MLLW; 303.2, -3 ft. MLLW, 303.3, -6 ft MLLW. At station 303.4 the transplanted mussels were hung from a floating dock, and were always approximately 8 ft. below sea level.

Figure 1

Sample Locations 1/6 - 1/7/98



APPENDIX B

ANALYTICAL RESULTS FROM
WATER AND TISSUE SAMPLES

QA/QC SUMMARY

PROJECT: Heckathorn Biomonitoring
PARAMETER: Pesticides and Total Lipids
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Tissues

SAMPLE CUSTODY: Eight mussel tissue samples were received on 1/9/98. All containers were received in good condition. The cooler temperature on arrival was 0.4°C. Samples were assigned a Battelle Central File (CF) identification number (1142) and were entered into Battelle's log-in system.

QA/QC DATA QUALITY OBJECTIVES:

<u>Analyte</u>	<u>Extraction Method</u>	<u>Analytical Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Achieved Detection Limit (ng/L)</u>
2,4'-DDE	MeCl ₂	GC-ECD	40-120%	±30%	0.26
Dieldrin	MeCl ₂	GC-ECD	40-120%	±30%	0.52
4,4'-DDE	MeCl ₂	GC-ECD	40-120%	±30%	0.19
2,4'-DDD	MeCl ₂	GC-ECD	40-120%	±30%	0.25
4,4'-DDD	MeCl ₂	GC-ECD	40-120%	±30%	0.26
2,4'-DDT	MeCl ₂	GC-ECD	40-120%	±30%	0.18
4,4'-DDT	MeCl ₂	GC-ECD	40-120%	±30%	0.15
Total Lipids	CHCl ₃	Gravimetric	NA	±30%	NA

METHOD: Chlorinated pesticides were analyzed according to a Battelle SOP based on EPA Method 8081 (EPA 1986) with modifications based on Krahn et al. (1988). Tissue samples were macerated and extracted with methylene chloride. Interferences were removed by aluminum/silicon column chromatography followed by high-performance liquid chromatography (HPLC) clean-up. Sample extracts were then transferred to cyclohexane and analyzed by capillary-column (DB-1701) gas chromatography with electron-capture detection (GC/ECD). Total lipids were determined according to the Bligh et al. (1959) method, modified to accommodate a smaller sample size. Lipids were extracted from separate aliquots of tissue samples using chloroform and the lipid weight obtained gravimetrically.

HOLDING TIMES: All extractions and analyses were conducted within target holding times: 14 days to extraction (refrigerated, not frozen), and 40 days to analysis after extraction. Samples were received on 1/9/98 and held at 4°C. Samples were extracted on 1/21/98, HPLC cleanup was conducted on 1/22/98, and GC analysis took place between 1/23/98 and 1/27/98. Lipid extractions were conducted on 1/22/98.

QA/QC SUMMARY

- DETECTION LIMITS:** Detection limits were determined by a previously conducted MDL study where replicates were analyzed and the standard deviation was multiplied by the Student's-t value for the number of replicates.
- BLANKS/BLANK SPIKES:** One procedural blank and two blank spikes were analyzed. All analytes were undetected in the blank. Blank spike recoveries of the two spiked analytes of interest, dieldrin and 4,4'-DDT, were within the target range of 40%-120%.
- REPLICATES:** One tissue sample 1142HB-19 (303.3T) was analyzed in duplicate. Precision for duplicate analyses are reported by calculating the relative percent difference (RPD) of replicate results. RPDs for all analytes of interest ranged from 0% to 8%, and were all within the QC limits of $\pm 30\%$.
- MATRIX SPIKES:** A matrix spike and matrix spike duplicate were analyzed using sample 1142HB-21(303.4T). Recoveries of the two spiked analytes of interest, dieldrin and 4,4'-DDT, were within the target range of 40%-120% in both the MS and MSD. The RPD between the MS and MSD was $<30\%$ for both dieldrin and 4,4'-DDT.
- REFERENCES:**
- Bligh, E.G., and W.J. Dyer. 1959. A Rapid Method of Total Lipid Extraction and Purification. *Canadian Journal of Biochemistry and Physiology*. 37:8 911-917.
- Krahn, M.M, CA Wigren, R.W. Pearce, S.K. Moore, R.G. Bogar, W. D. McLeod, Jr., S.L. Chan, and D.W. Brown. 1988. *New HPLC Cleanup and Revised Extraction Procedures for Organic Contaminants*. NOAA Technical Memorandum MNFS F/NWC-153. Standard Analytical Procedures of the NOAA National Facility, 1988. National Oceanic and Atmospheric Administration, National Marine Fisheries Service, Seattle, WA.
- U.S. EPA. 1986 (Revised 1990). *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

BATTELLE MARINE SCIENCES LABORATORY

1529 West Sequim Bay Road

Sequim, WA 98382-9099

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Pesticides in Tissue

Samples Received 1/9/98

MSL Code	1142HB*1	1142HB*17	1142HB*18	1142HB*19	1142HB*20	1142HB*21	1142HB*22	1142HB*23	1142HB*24
Sponsor ID	Background	303.1T	303.1R	303.3T	303.3R	303.4T	303.4R	303.2T	303.2R
Matrix	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue	Tissue
Wet Wt (g)	20.1	20.1	20.2	20.1	20.5	20.2	20.6	20.1	20.4
Percent Dry Wt	15.2	11.8	10.8	10.9	10.6	11.9	8.94	10.8	8.90
Extraction Date	01/21/98	01/21/98	01/21/98	01/21/98	01/21/98	01/21/98	01/21/98	01/21/98	01/21/98
Analytical Batch	1	1	1	1	1	1	1	1	1
Lipids (%dry wt)	9.60	9.61	9.57	8.71	9.94	10.1	6.26	9.99	10.2
Unit (wet wt)	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g	ng/g
2,4 DDE	0.26 U	0.26 U	0.26 U	19.5 D	29.5 D	4.46 D	3.03 D	8.22 D	10.2 D
DIELDRIN	0.66	11.2 D	5.43	279 DD	184 D	83.1 D	8.18	165 D	40.3 D
4,4 DDE	0.67	11.7 D	19.4 D	207 DD	434 DD	54.2 D	29.9 D	119 D	107 DD
2,4 DDD	0.25 U	17.2 D	20.4 D	702 DD	820 DD	138 D	47.1 D	272 D	203 DD
4,4 DDD	0.38	70.5 D	66.4 D	1980 DD	1860 DD	349 DD	136 D	825 DD	589 DD
2,4 DDT	0.18 U	4.52	7.46	220 DD	549 DD	26.2 D	15.0 D	81.2 D	145 D
4,4 DDT	0.15 U	9.04	13.6	373 DD	811 DD	41.3 D	24.7 D	143 D	168 DD
SURROGATE RECOVERIES (%)									
PCB103	90.8	73.5	73.0	124 D	108 D	117 D	81.5	94.6 D	92.1 D
PCB198	88.4	87.3	85.9	123 D	99.8 D	124 D	93.2	113 D	103 D
PCB103	NA	90.5 D	82.7 D	144 DD	116 DD	160 DD	115 D	137 DD	110 DD
PCB198	NA	112 D	101 D	137 DD	113 DD	141 DD	123 D	132 DD	129 DD

D 9X Dilution

DD 100X Dilution

U Not detected at or above DL shown

Outside QA/QC limits of 40-120%

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Pesticides in Tissue

Samples Received 1/9/98

MSL Code Sponsor ID	BSA				BSB			DUP		RPD
	Blank	Blank Spike A	Spike Amount	Percent Recovery	Blank Spike B	Spike Amount	Percent Recovery	1142HB*19 303.3 Trans	1142HB*19 303.3 Trans	
Matrix	Tissue	Tissue			Tissue			Tissue	Tissue	
Wet Wt (g)	NA	NA			NA			20.1	20.1	
Percent Dry Wt	NA	NA			NA			10.9	10.9	
Extraction Date	01/21/98	01/21/98			01/21/98			01/21/98	01/21/98	
Analytical Batch	1	1			1			1	1	
Lipids (%dry wt)	NA	NA			NA			8.71	8.71	
Unit (wet wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	ng/g	ng/g	%
2,4 DDE	0.26 U	0.26 U	NS	NA	0.26 U	NS	NA	19.5 D	19.2 D	2%
DIELDRIN	0.52 U	4.57	5.00	91%	4.30	5.00	86%	279 DD	257 DD	8%
4,4 DDE	0.19 U	0.19 U	NS	NA	0.19 U	NS	NA	207 DD	207 DD	0%
2,4 DDD	0.25 U	0.25 U	NS	NA	0.25 U	NS	NA	702 DD	711 DD	1%
4,4 DDD	0.26 U	0.26 U	NS	NA	0.26 U	NS	NA	1980 DD	1980 DD	0%
2,4 DDT	0.18 U	0.18 U	NS	NA	0.18 U	NS	NA	220 DD	219 DD	0%
4,4 DDT	0.15 U	5.24	5.00	105%	6.02	5.00	120%	373 DD	371 DD	1%

SURROGATE RECOVERIES (%)

PCB103	90.1	107			97.2			124 #D	127 #D	
PCB198	93.4	113			103			123 #D	123 #D	
PCB103								144 #DD	148 #DD	
PCB198								137 #DD	140 #DD	

D 9X Dilution

DD 100X Dilution

U Not detected at or above DL shown

Outside QA/QC limits of 40-120%

Note: PCB 198 is surrogate for the target analytes; target analyte concentrations are corrected using the PCB 198 percent recovery.

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 Pesticides in Tissue
 Samples Received 1/9/98

MSL Code	MSA		Spike Amount	Percent Recovery	MSB		Spike Amount	Percent Recovery	RPD
	1142HB*21	1142HB*21			1142HB*21				
Sponsor ID	303.4T	Spike A			Spike B				
Matrix	Tissue	Tissue			Tissue				
Wet Wt (g)	20.2	20.0			20.4				
Percent Dry Wt	11.9	11.9			11.9				
Extraction Date	01/21/98	01/21/98			01/21/98				
Analytical Batch	1	1			1				
Lipids (%dry wt)	10.1	10.1			10.1				
Unit (wet wt)	ng/g	ng/g	ng/g	%	ng/g	ng/g	%	%	
2,4 DDE	4.46 D	5.07 D	NS	NA	5.60 D	NS	NA		
DIELDRIN	83.1 D	85.9 D	5.00	56%	86.6 D	4.90	71%	24%	
4,4 DDE	54.2 D	52.7 D	NS	NA	54.2 D	NS	NA		
2,4 DDD	138 D	135 D	NS	NA	141 D	NS	NA		
4,4 DDD	349 DD	332 DD	NS	NA	352 DD	NS	NA		
2,4 DDT	26.2 D	7.01 D	NS	NA	26.5 D	NS	NA		
4,4 DDT	41.3 D	44.7 D	5.00	68%	45.3 D	4.90	82%	18%	

SURROGATE RECOVERY

PCB103	117 D	115 D	106 D
PCB198	124 #D	120 D	111 D
PCB103			
PCB198			

D 9X Dilution
 DD 100X Dilution
 U Not detected at or above
 # Outside QA/QC limits
 Note: PCB 198 is surrogate

QA/QC SUMMARY

PROJECT: Heckathorn Biomonitoring
PARAMETER: Pesticides
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Water

SAMPLE CUSTODY: Fifteen water samples in three coolers were received on 1/9/98. All containers were received in good condition. Cooler temperatures upon arrival were as follows:
Sample 303.1 (3 replicates): 4.2°C
Samples 303.3, 303.2 (3 replicates of each), 303.5: 1.9°C
Sample 303.4 (3 replicates, MS, MSD): 2.1°C
Samples were assigned a Battelle Central File (CF) identification number (1142HB) and were entered into Battelle's log-in system.

QA/QC DATA QUALITY OBJECTIVES:

<u>Analyte</u>	<u>Extraction Method</u>	<u>Analytical Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Achieved Detection Limit (ng/L)</u>
2,4'-DDE	MeCl ₂	GC-ECD	40-120%	±30%	0.03
Dieldrin	MeCl ₂	GC-ECD	40-120%	±30%	0.01
4,4'-DDE	MeCl ₂	GC-ECD	40-120%	±30%	0.03
2,4'-DDD	MeCl ₂	GC-ECD	40-120%	±30%	0.11
4,4'-DDD	MeCl ₂	GC-ECD	40-120%	±30%	0.05
2,4'-DDT	MeCl ₂	GC-ECD	40-120%	±30%	0.05
4,4'-DDT	MeCl ₂	GC-ECD	40-120%	±30%	0.04

METHOD: Chlorinated pesticides were analyzed according to a Battelle SOP based on EPA Method 8081 (EPA 1986). Water samples were extracted with methylene chloride. Interferences were removed by aluminum/silicon column chromatography. Sample extracts were then transferred to cyclohexane and analyzed by capillary-column gas chromatography with electron-capture detection (GC/ECD).

HOLDING TIMES: All extractions and analyses were conducted within target holding times: 14 days to extraction, and 40 days to analysis after extraction. Samples were received on 1/9/98 and held at 4°C. Samples were extracted on 1/12/98 and 1/14/98 and analyzed between 1/15/98 and 1/22/98.

DETECTION LIMITS: Detection limits were determined by a previously conducted MDL study where replicates were analyzed and the standard deviation was multiplied by the Student's-t value for the number of replicates.

SURROGATES: Recoveries of one or both of the two surrogate compounds (PCB 103 and PCB 198) exceeded data quality limits of 40%-120% in eight of the samples and in four of the QC samples. Samples 1142HB-9 (303.4) and 1142HB-15 (303.2) required additional drying and evaporation steps to remove residual water. This extra handling of the samples could account for the low recoveries of both surrogates. Recoveries of both surrogates in samples 1142HB-7 (303.3) and 1142HB-13 MSD (303.4) were low, possibly due to lab mishaps where portions of the samples may have been lost. The other samples had at least one surrogate (PCB 198) within recovery criteria. Because the more volatile of the two surrogates was lost, the low recovery may be due to extra

QA/QC SUMMARY

evaporation steps to achieve a low final sample volume of 0.2 mL. All data were surrogate-corrected; therefore, the low recoveries do not affect the reported analyte data.

BLANKS/BLANK SPIKES:

Two procedural blanks were analyzed. All analytes except 4,4'-DDE in Blank 1 were undetected. Blank spike recoveries of the two spiked analytes of interest, dieldrin and 4,4'-DDT, were within the target range of 40%-120%.

MATRIX SPIKES:

A matrix spike and matrix spike duplicate were analyzed using the samples provided (two additional replicates of sample no. 303.4). Recoveries of the two spiked analytes of interest, dieldrin and 4,4'-DDT, were within the target range of 40%-120% in the MS. In the MSD, dieldrin was within the QC range, but 4,4'-DDT exceeded recovery criteria (129%).

REPLICATES:

Four water samples were provided in triplicate, with an additional blind duplicate of sample 303.3 (sample 303.5). Precision for triplicate analyses is reported by calculating the relative standard deviation (RSD) of replicate results. RSDs for all analytes of interest were variable, ranging from 2% to 82%.

REFERENCES:

U.S. EPA. 1986 (Revised 1990). *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846*. 3rd ed. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

BATTELLE MARINE SCIENCES LABORATORY

1529 West Sequim Bay Road

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Pesticides in Water

Samples Received 1/9/98

MSL Code	1142HB*2	1142HB*3	1142HB*4	1142HB*5	1142HB*6	1142HB*7	1142HB*8
Sponsor ID	303.1	303.1	303.1	303.3	303.3	303.3	303.5
Matrix	Water	Water	Water	Water	Water	Water	Water
Extracted Volume (mL)	1830	1850	1810	1830	1850	1850	1850
Extraction Date	01/12/98	01/12/98	01/12/98	01/12/98	01/12/98	01/12/98	01/12/98
Analytical Batch	1	1	1	1	1	1	1
Unit	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
2,4 DDE	0.02 U	0.02 U	0.03 U	0.37	0.39	1.07	0.02 U
DIELDRIN	0.47	1.16	0.32	14.7	22.5	17.1 D	23.3 D
4,4 DDE	0.15 B	0.17 B	0.17 B	8.57	15.7	43.5 D	9.78
2,4 DDD	0.28	0.44	0.10 U	16.7	17.6	6.98	20.9 D
4,4 DDD	0.05 U	0.05 U	0.52	31.3 D	27.8 D	2.28	54.6 D
2,4 DDT	0.05 U	0.05 U	0.22	7.79	10.6	7.80	16.0
4,4 DDT	0.04 U	0.04 U	0.04 U	27.0 D	65.9 D	19.1	93.7 D
<u>SURROGATE RECOVERIES (%)</u>							
PCB103	55.9	47.4	52.3	64.8	86.6	34.5 #	57.3
PCB198	78.8	65.8	74.9	77.3	103	42.9	70.9

B Analyte detected in sample is <5x blank concentration

D 5X Dilution

U Not detected at or above DL shown

Outside QA/QC limits of 40-120%

Note: PCB 198 is surrogate for the target analytes; target analyte concentrations are corrected using the PCB 198 percent recovery.

BATTELLE MARINE SCIENCES LABORATORY

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Pesticides in Water

Samples Received 1/9/98

MSL Code	1142HB*9	1142HB*10	1142HB*11	1142HB*12	1142HB*13	1142HB*14	1142HB*15	1142HB*16
Sponsor ID	303.4	303.4	303.4	303.4 MS A	303.4 MS B	303.2	303.2	303.2
Matrix	Water	Water	Water	Water	Water	Water	Water	Water
Extracted Volume (mL)	1840	1870	1860	1850	1590	1880	1890	1850
Extraction Date	01/14/98	01/14/98	01/14/98	01/14/98	01/14/98	01/14/98	01/14/98	01/14/98
Analytical Batch	1	1	1	1	1	1	1	1
Unit	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
2,4 DDE	0.02 U	0.02 U	0.02 U	0.02 U	0.03 U	0.02 U	0.02 U	0.02 U
DIELDRIN	2.65	2.16	2.61	7.48	7.39	8.84	10.3	5.40
4,4 DDE	0.03 U	0.03 U	0.03 U	1.05	1.09	2.25	2.24	2.34
2,4 DDD	2.01	2.04	2.26	3.03	2.46	6.60	6.31	6.06
4,4 DDD	9.62	8.07	8.84	8.88	9.29	19.7	18.6	14.8
2,4 DDT	0.10	0.15	0.05 U	0.05 U	0.08	3.90	3.69	3.72
4,4 DDT	0.04 U	0.04 U	0.04 U	6.28	8.08	12.6	15.6	9.33
<u>SURROGATE RECOVERIES (%)</u>								
PCB103	26.3 #	39.9 #	37.3 #	51.3	19.3 #	50.8	27.9 #	43.3
PCB198	33.9	49.6	45.6	64.3	24.6 #	61.9	41.9	48.6

B Analyte detected in sample is <5x blank concentration

D 5X Dilution

U Not detected at or above DL shown

Outside QA/QC limits of 40-120%

Note: PCB 198 is surrogate for the target analytes; target analyte concentrations are corrected using the PCB 198 percent recovery.

BATTELLE MARINE SCIENCES LABORATORY

1529 West Sequim Bay Road

Sequim, WA 98382-9099

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HECKATHORNPesticides in Water
Samples Received 1/9/98

MSL Code Sponsor ID	BSA				BSB			
	Blank	Blank Spike A	Spike Amount	Percent Recovery	Blank Spike B	Spike Amount	Percent Recovery	Blank2
Matrix	Water	Water			Water			Water
Extracted Volume (mL)	1700	1700			1700			1800
Extraction Date	01/12/1998	01/12/1998			01/12/1998			01/14/1998
Analytical Batch	1	1			1			1
Unit	ng/L	ng/L	ng/L	%	ng/L	ng/L	%	ng/L
2,4 DDE	0.03 U	0.03 U	NS	NA	0.03 U	NS	NA	0.03 U
DIELDRIN	0.01 U	6.61	5.88	112%	5.10	5.88	87%	0.01 U
4,4 DDE	0.11	0.65	NS	NA	0.96	NS	NA	0.03 U
2,4 DDD	0.11 U	0.11 U	NS	NA	0.11 U	NS	NA	0.11 U
4,4 DDD	0.05 U	0.05 U	NS	NA	0.05 U	NS	NA	0.05 U
2,4 DDT	0.05 U	0.05 U	NS	NA	0.05 U	NS	NA	0.05 U
4,4 DDT	0.05 U	6.03	5.88	103%	6.09	5.88	104%	0.04 U

SURROGATE RECOVERIES (%)

PCB103	47.5	58.2		44.2		50.2
PCB198	56.2	81.5		71.4		70.9

D 5X Dilution

U Not detected at or above DL shown

Outside QA/QC limits of 40-120%

Note: PCB 198 is surrogate for the target analytes; target analyte concentrations are corrected using the PCB 198 percent recovery.

BATTELLE MARINE SCIENCES LABORATORY

1529 West Sequim Bay Road

Sequim, WA 98382-9099

360/681-3643

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Pesticides in Water

Samples Received 1/9/98

	MSA				MSB		
MSL Code	1142-11	1142-12	Spike	Percent	1142-13	Spike	Percent
Sponsor ID		Spike A	Amount	Recovery	Spike B	Amount	Recovery
Matrix	Water	Water			Water		
Extracted Volume (mL)	1860	1850			1590		
Extraction Date	01/14/1998	01/14/1998			01/14/1998		
Analytical Batch	1	1			1		
Unit	ng/L	ng/L	ng/L	%	ng/L	ng/L	%
2,4 DDE	0.02 U	0.02 U	NS	NA	0.03 U	NS	NA
DIELDRIN	2.61	7.48	5.40	90%	7.39	6.28	76%
4,4 DDE	0.03 U	1.05	NS	NA	1.09	NS	NA
2,4 DDD	2.26	3.03	NS	NA	2.46	NS	NA
4,4 DDD	8.84	8.88	NS	NA	9.29	NS	NA
2,4 DDT	0.05 U	0.05 U	NS	NA	0.08	NS	NA
4,4 DDT	0.04 U	6.28	5.40	116%	8.08	6.28	129% #

SURROGATE RECOVERIES (%)

PCB103	37.3 #	51.3	19.3 #
PCB198	45.6	64.3	24.6 #

D 5X Dilution

U Not detected at or above DL shown

Outside QA/QC limits of 40-120%

Note: PCB 198 is surrogate for the target analytes; target analyte concentrations are corrected using the PCB 198 percent recovery.

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